

like cinder, which it was necessary to remove in order to obtain sufficient draft. It therefore appears, that although samples of this fuel can be selected which will give favorable results upon the small scale, the mass of the vein can hardly be used for the general purposes to which anthracite is applicable, and that it requires some especial device for the removal of the voluminous ash, to enable the successful and continuous use of the fuel for ordinary purposes.

CONTRIBUTIONS FROM THE LABORATORY OF THE UNIVERSITY OF PENNSYLVANIA.

No. XII.

A new method for the Decomposition of Chromic Iron. By Edgar F. Smith, Ph. D., Assistant in Analytical Chemistry, University of Pennsylvania.

(Read before the American Philosophical Society, December 21, 1877.)

Recently I was led to try the action of bromine and sodium hydrate upon pulverized chromic iron, and as the amount of chromium extracted in this manner was rather surprising, the following experiments were made, to ascertain what effect bromine alone in presence of water would have upon the same substance.

I. Moderately fine chromic iron (.1500 Grm.) was placed in a tube of hard glass, and after adding dilute bromine water and sealing the tube, the latter was placed in an air-bath and heated for twelve hours at a temperature of about 130° C.; when cool the tube was opened and its contents poured upon a filter. The insoluble residue was thoroughly washed by decantation, and upon the filter, with hot water. The filtrate after concentration was treated with a slight excess of ammonium hydrate, causing the precipitation of aluminum hydrate, &c. The latter was filtered off and the yellow colored filtrate, then warmed with hydrogen sulphide to reduce the chromic acid to oxide. The precipitate formed, after protracted digestion, was allowed to settle and the clear liquid filtered. After washing the precipitate it was dissolved in a few drops of dilute hydrochloric acid and re-precipitated. This operation was repeated and the precipitate finally transferred to a filter washed, dried and ignited. The amount of chromium oxide found corresponded to 15.50 per cent, of the substance taken.

The amount of chromium remaining in the material not attacked by the bromine was not estimated.

II. .2000 grms, substance, as finely pulverized as could be obtained by grinding the material in an agate mortar, were heated in a sealed tube with water saturated with bromine and a few drops of bromine. The tube was allowed to remain in the oven for four days, the temperature ranging from 175° - 190° C. Upon opening the tube its contents were poured into a

beaker and evaporated; water then added and the solution filtered. The residual, unattacked mineral powder after washing, drying and igniting, weighed .0820 grms. The filtrate from this was treated precisely as in (I) and the chromium oxide obtained from it amounted to 28.05 per cent.

III. In this experiment only .1500 grms. substance were employed. The material was of the same fineness as in (II). Instead of using dilute bromine water as heretofore, an excess of bromine was poured over the substance and but a very small quantity of water added. For three days the tube was exposed to a temperature varying from 150° - 175° C. At the expiration of this time the tube was examined, and as the substance appeared to be perfectly decomposed, the solution was removed from the tube and evaporated in a beaker glass to expel the large excess of bromine, upon the gradually disappearance of which a dark powder showed itself. The solution was strongly diluted with water and filtered. The insoluble residue was thoroughly washed with hot water. Dried and ignited, this weighed .0140 grms.

The filtrate was mixed with an excess of ammonium hydrate and evaporated almost to dryness in a casserole. The solution was then diluted with water and filtered from the aluminum hydrate, &c., and treated as in (I). The percentage of chromium oxide extracted equaled 49.60 per cent.

IV. From the preceding experiments it appeared very evident, that all that was lacking to render the decomposition complete was to have the chromic iron in an exceedingly fine condition. To this end the material that had been ground to an impalpable powder in an agate mortar was elutriated, then dried, and two separate portions of .1500 grms. each placed in good, hard glass tubes. To each portion was added a rather large quantity of bromine water and from 10-12 drops of bromine. Both tubes were heated for one day at 130° C. For two successive days the temperature was maintained at 170° C. At the expiration of the third day, one of the tubes was removed from the oven and opened. Red oxide of iron had separated and undecomposed material was no longer visible. The whole was poured into a beaker and evaporated; water added and filtered. The residue was thoroughly washed, dried and ignited, then transferred to a beaker glass and heated with dilute hydrochloric acid. The entire mass dissolved readily and without a residue. The decomposition was, therefore, complete.

The filtrate from the ferric oxide was evaporated almost to dryness after the addition of an excess of ammonium hydrate, then diluted and filtered. The solution was reduced with hydrogen sulphide and the precipitate, after filtering and washing, dissolved in dilute hydrochloric acid and re-precipitated with ammonium hydrate. This operation was repeated and the chromium oxide obtained was 62.66%.

The second tube which was removed not long after the first, contained a large amount of separated ferric oxide. This, after filtering off the chromium solution, also dissolved very readily in warm, dilute hydrochloric acid, leaving not the least trace of residue.

The filtrate from this, after being similarly treated as above, yielded 62.83 per cent. chromium oxide.

These results accord with those of Garrett, who analyzed the same ore from Texas, Pa., and obtained about 63. per cent. of chromium oxide.

The ferric oxide that separates out in the tube during the decomposition will not contain any chromium whatever, if it is thoroughly washed with boiling water.

In no instance was iron found in the solution containing the chromic acid.

Several tubes containing the pulverized substance, potassium hydrate and bromine water, were heated at 125° C., but invariably exploded before the decomposition was completed, and therefore no further attempts were made to use the alkali to aid in the decomposition.

All that is necessary to effect the complete decomposition of chromic iron by this method is that the substance be exceedingly fine, and that the same be exposed with bromine water to a temperature of 180° C., from two to three days. The addition of 10-12 drops of bromine hastens the decomposition very decidedly.

In connection with the above, it may be well to mention that the insoluble chromium oxide obtained by the ignition of the corresponding hydrate, may be brought into solution again by digesting it together with bromine and sodium hydrate in a beaker.

Precipitation of Copper with Sodium Carbonate.

The precipitation of copper from its solutions by sodium hydrate, gives a precipitate that is worked with difficulty. Sodium carbonate, on the other hand, added to similar solutions, and these boiled, affords a dark brown, granular precipitate, that may be readily and completely washed with hot water.

Mr. Harry G. McCarter, student in the Laboratory of the University, made the following analyses, which show that the method yields as accurate results as could be desired.

I. .2000 grms. $\text{CuSO}_4 + 5\text{H}_2\text{O}$ —dissolved in water and precipitated with sodium carbonate, gave .0630 gm., $\text{CuO} = 25.15$ per cent Cu.

II. .2000 grms. $\text{CuSO}_4 + 5\text{H}_2\text{O}$ —treated as above, gave .0634 gm. $\text{CuO} = 25.30$ per cent Cu.

The theoretical percentage of copper in the salt is 25.25 per cent.

After the addition of sodium carbonate in slight excess to the copper salt solution, the latter was boiled for an hour, until all the carbon dioxide was expelled.

The filtrates from the precipitates in every instance were evaporated, but not the slightest trace of copper discovered.

An excess of acid in the solution from which it is desired to precipitate copper by an alkaline carbonate, should be avoided.

The presence of rather large quantities of alkaline nitrates or sulphates will cause the solution of the precipitates first produced by the carbonates. Continued boiling will not remedy the matter. From such solutions, however, the alkaline hydrates will not fail to precipitate the copper.